

09/12/00  
3c922 U.S. PRO

09-13-00

A

Please type a plus sign (+) inside this box → ☐

PTO/SB/05 (4/98)  
Approved for use through 09/30/2000. OMB 0651-0032  
Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

# UTILITY PATENT APPLICATION TRANSMITTAL

Attorney Docket No. DB000817-000  
First Inventor or Application Identifier Keith Henry  
Title WATER DILUTED CUMENE HYDROPEROXIDE SOLUTIONS  
Express Mail Label No. EJ116542545US

## APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

ADDRESS TO: Assistant Commissioner for Patents  
Box Patent Application  
Washington, DC 20231

- ☒ \* Fee Transmittal Form (e.g., PTO/SB/17)  
(Submit an original and a duplicate for fee processing)
- ☒ Specification [Total Pages 12]  
(preferred arrangement set forth below)
  - Descriptive title of the Invention
  - Cross References to Related Applications
  - Statement Regarding Fed sponsored R & D
  - Reference to Microfiche Appendix
  - Background of the Invention
  - Brief Summary of the Invention
  - Brief Description of the Drawings (if filed)
  - Detailed Description
  - Claim(s)
  - Abstract of the Disclosure
- ☒ Drawing(s) (35 U.S.C. 113) [Total Sheets 4]
- Oath or Declaration [Total Pages 3]
  - ☒ Newly executed (original or copy)
  - ☐ Copy from a prior application (37 C.F.R. § 1.63(d))  
(for continuation/divisional with Box 16 completed)
    - ☐ **DELETION OF INVENTOR(S)**  
Signed statement attached deleting inventor(s) named in the prior application, see 37 C.F.R. §§ 1.63(d)(2) and 1.33(b).

- ☐ Microfiche Computer Program (Appendix)
- Nucleotide and/or Amino Acid Sequence Submission (if applicable, all necessary)
  - ☐ Computer Readable Copy
  - ☐ Paper Copy (identical to computer copy)
  - ☐ Statement verifying identity of above copies

## ACCOMPANYING APPLICATION PARTS

- ☒ Assignment Papers (cover sheet & document(s))
- ☒ 37 C.F.R. § 3.73(b) Statement of Attorney (when there is an assignee) ☒ Power of Attorney
- ☐ English Translation Document (if applicable)
- ☒ Information Disclosure Statement (IDS)/PTO-1449 ☒ Copies of IDS Citations
- ☐ Preliminary Amendment
- ☒ Return Receipt Postcard (MPEP 503)  
(Should be specifically itemized)
- ☐ \* Small Entity Statement(s) ☐ Statement filed in prior application, Status still proper and desired (PTO/SB/09-12)
- ☐ Certified Copy of Priority Document(s) (if foreign priority is claimed)
- ☐ Other:

\* NOTE FOR ITEMS 1 & 13: IN ORDER TO BE ENTITLED TO PAY SMALL ENTITY FEES, A SMALL ENTITY STATEMENT IS REQUIRED (37 C.F.R. § 1.27), EXCEPT IF ONE FILED IN A PRIOR APPLICATION IS RELIED UPON (37 C.F.R. § 1.28).

16. If a CONTINUING APPLICATION, check appropriate box, and supply the requisite information below and in a preliminary amendment:  
☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No: \_\_\_\_\_  
Prior application information: Examiner \_\_\_\_\_ Group / Art Unit: \_\_\_\_\_

For CONTINUATION or DIVISIONAL APPS only: The entire disclosure of the prior application, from which an oath or declaration is supplied under Box 4b, is considered a part of the disclosure of the accompanying continuation or divisional application and is hereby incorporated by reference. The incorporation can only be relied upon when a portion has been inadvertently omitted from the submitted application parts.

## 17. CORRESPONDENCE ADDRESS

☐ Customer Number or Bar Code Label (Insert Customer No. or Attach bar code label here) or ☒ Correspondence address below

Name	John E. Mrozinski, Jr.				
	Thorp Reed & Armstrong, LLP				
Address	One Riverfront Center				
City	Pittsburgh	State	PA	Zip Code	15222-2611
Country	United States	Telephone	412-394-2320	Fax	412-394-2555

Name (Print/Type)	John E. Mrozinski, Jr.	Registration No. (Attorney/Agent)	46,179
Signature	<i>John E. Mrozinski Jr.</i>	Date	September 12, 2000

Burden Hour Statement: This form is estimated to take 0.2 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Box Patent Application, Washington, DC 20231.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

# FEE TRANSMITTAL

## for FY 2000

Patent fees are subject to annual revision.

Small Entity payments must be supported by a small entity statement, otherwise large entity fees must be paid. See Forms PTO/SB/09-12.

See 37 C.F.R. §§ 1.27 and 1.28.

TOTAL AMOUNT OF PAYMENT (\$) 1012.00

## Complete if Known

Application Number	Not yet assigned
Filing Date	September 12, 2000
First Named Inventor	Henry et al.
Examiner Name	Not yet assigned
Group / Art Unit	Not yet assigned
Attorney Docket No.	DB000817-000

09/12/00  
09/660099  
JCE41 U.S. Pat. & Tm. Off.

## METHOD OF PAYMENT (check one)

- 1.
- ☒
- The Commissioner is hereby authorized to charge indicated fees and credit any overpayments to:

Deposit Account Number 20-0888

Deposit Account Name

☒ Charge Any Additional Fee Required Under 37 CFR §§ 1.16 and 1.17

- 2.
- ☒
- Payment Enclosed:

☒ Check ☐ Money Order ☐ Other

## FEE CALCULATION

## 1. BASIC FILING FEE

Large Entity Fee Code (\$)	Small Entity Fee Code (\$)	Fee Description	Fee Paid
101 690	201 345	Utility filing fee	690
106 310	206 155	Design filing fee	
107 480	207 240	Plant filing fee	
108 690	208 345	Reissue filing fee	
114 150	214 75	Provisional filing fee	

SUBTOTAL (1) (\$) 690

## 2. EXTRA CLAIM FEES

Total Claims	Extra Claims	Fee from below	Fee Paid
27	-20** = 7	18	126
5	-3** = 2	78	156
Multiple Dependent			

\*\*or number previously paid, if greater; For Reissues, see below

## Large Entity Small Entity

Large Entity Fee Code (\$)	Small Entity Fee Code (\$)	Fee Description
103 18	203 9	Claims in excess of 20
102 78	202 39	Independent claims in excess of 3
104 260	204 130	Multiple dependent claim, if not paid
109 78	209 39	** Reissue independent claims over original patent
110 18	210 9	** Reissue claims in excess of 20 and over original patent

SUBTOTAL (2) (\$) 282

## FEE CALCULATION (continued)

## 3. ADDITIONAL FEES

Large Entity Fee Code (\$)	Small Entity Fee Code (\$)	Fee Description	Fee Paid
105 130	205 65	Surcharge - late filing fee or oath	
127 50	227 25	Surcharge - late provisional filing fee or cover sheet	
139 130	139 130	Non-English specification	
147 2,520	147 2,520	For filing a request for reexamination	
112 920*	112 920*	Requesting publication of SIR prior to Examiner action	
113 1,840*	113 1,840*	Requesting publication of SIR after Examiner action	
115 110	215 55	Extension for reply within first month	
116 380	216 190	Extension for reply within second month	
117 870	217 435	Extension for reply within third month	
118 1,360	218 680	Extension for reply within fourth month	
128 1,850	228 925	Extension for reply within fifth month	
119 300	219 150	Notice of Appeal	
120 300	220 150	Filing a brief in support of an appeal	
121 260	221 130	Request for oral hearing	
138 1,510	138 1,510	Petition to institute a public use proceeding	
140 110	240 55	Petition to revive - unavoidable	
141 1,210	241 605	Petition to revive - unintentional	
142 1,210	242 605	Utility issue fee (or reissue)	
143 430	243 215	Design issue fee	
144 580	244 290	Plant issue fee	
122 130	122 130	Petitions to the Commissioner	
123 50	123 50	Petitions related to provisional applications	
126 240	126 240	Submission of Information Disclosure Stmt	
581 40	581 40	Recording each patent assignment per property (times number of properties)	40
146 690	246 345	Filing a submission after final rejection (37 CFR § 1.129(a))	
149 690	249 345	For each additional invention to be examined (37 CFR § 1.129(b))	
Other fee (specify)			
Other fee (specify)			

\* Reduced by Basic Filing Fee Paid

SUBTOTAL (3) (\$) 40

## SUBMITTED BY

Name (Print/Type)	John E. Mrozinski, Jr.	Registration No. (Attorney/Agent)	46,179	Telephone	412-394-2320
Signature	<i>John E. Mrozinski, Jr.</i>	Date	September 12, 2000		

## WARNING:

Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

Burden Hour Statement: This form is estimated to take 0.2 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

EXPRESS MAIL MAILING LABEL

Number: EJ116542545US

Date of Deposit: September 12, 2000

I hereby certify that this paper or fee is being deposited with the United States Postal Service under 37 C.F.R. § 1.10 on the date indicated above and is addressed to the Commissioner for Patents, Washington, D.C. 20231

THORP REED & ARMSTRONG, LLP  
One Riverfront Center  
Pittsburgh, PA 15222-4895



*Danica Mannion*

**PATENT**

**Attorney Docket No.: DB000817-000**

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

**Applicant:** Henry et al. )  
Serial No.: Not yet assigned )  
**Filed:** September 12, 2000 )  
**Entitled:** WATER DILUTED CUMENE HYDROPEROXIDE SOLUTIONS )

**TRANSMITTAL LETTER FOR PATENT APPLICATION**

**To:** Commissioner for Patents  
Box PATENT APPLICATION  
Washington, D.C. 20231

Dear Sir:

Enclosed for filing is the above-referenced patent application. Transmitted herewith for filing are:

1. Utility Patent Application Transmittal (Form PTO/SB/05);
2. Fee Transmittal (Form PTO/SB/17), 2 copies and a check in the amount of \$1,012.00;
3. Twelve (12) pages of specification including Twenty-seven (27) claims;
4. Four (4) sheets of figures;
5. Declaration for Patent Application;
6. 37 C.F.R. § 3.73(b) Statement and Power of Attorney;
7. Recordation Form Cover Sheet;
8. Assignment from inventors to assignee;
9. Information Disclosure Statement; and
10. Form PTO/SB/08A and copies of nine (9) cited references.

Also enclosed is a return postcard. Please date stamp and mail the postcard to acknowledge receipt of the above-mentioned documents.

The Commissioner is hereby authorized to charge any underpayment or credit any overpayment to our Deposit Account No. 20-0888. A duplicate copy of this Transmittal is enclosed herewith.

Respectfully submitted,



John E. Mrozinski, Jr.

Thorp Reed & Armstrong, LLP

One Riverfront Center

Pittsburgh, PA 15222-4895

(412) 394-2320

Dated: September 12, 2000

Attorneys for Applicant

00254553

**WATER DILUTED CUMENE HYDROPEROXIDE SOLUTIONS**

**Field of Invention**

This invention relates in general to methods of reducing hazardous air pollutants in cumene hydroperoxide and improving the shipping and handling of cumene hydroperoxide in cold weather and more specifically, to the partial or total replacement of cumene as a diluent by water.

**Background of the Invention**

Cumene hydroperoxide (CHP) is produced as an intermediate product in the manufacture of phenol from cumene and oxygen. In the BP Chemicals/Hercules and similar phenol processes, cumene is oxidized to CHP which can then be cleaved with sulfuric acid to yield phenol and acetone. These processes account for nearly all the phenol produced in the world today and are described in numerous patents and published literature dating back to the early 1950's. CHP synthesis yields small amounts of the following which are considered impurities: cumene,  $\alpha$ -methylstyrene (AMS), dimethyl benzyl alcohol (DMBA) and acetophenone (AP). Further processing steps such as those described in U.S. Pat. No. 4,654,124, assigned to the same assignee as the present invention and incorporated in its entirety herein by reference, are necessary to remove these impurities and color formers from the CHP. Such purification as by the methods of the '124 patent may result in a CHP solution with a purity of greater than 92 weight percent.

Under United States federal regulations, 90 weight percent CHP is the maximum concentration permitted for domestic transport (49 C.F.R., Sec. 173.225, Organic Peroxide Table-revised October 1, 1998). Therefore, refined CHP must be diluted, typically with cumene, to a concentration of 82 weight percent-90 weight percent to produce commercial grades of CHP that can be transported in compliance with these regulations.

The use of cumene as a diluent of CHP solutions presents several problems for CHP producers, transporters and end users. First, because the U.S. Clean Air Act Amendments (Public Law 101-549 Sect. 112(b)) classify the diluent, cumene, as a hazardous air pollutant, precautions must be taken when producing, transporting and using CHP solutions to minimize cumene emissions. Second, because cumene can be considered an impurity in CHP solutions, it may have to be removed by the end user, thereby creating additional hazardous emissions problems. Third, if the cumene added to CHP solutions is not removed, it can result in the end user's finished product containing unacceptable impurities. Fourth, the climatic conditions at the end user's facility may vary and necessitate changing the concentration of purified CHP utilized. A lower concentration CHP solution, i.e., higher cumene, typically is used to prevent the CHP solution from freezing in colder months, but a higher concentration

CHP solution, i.e., lower cumene, is used in warmer months. Finally, the presence of cumene in CHP solutions may result in a reduced yield in the end user's process(es). The Inventors are unaware of any diluent, other than cumene, that is being used commercially with CHP.

Therefore, a need exists in the art for a CHP-diluent solution which will reduce or eliminate the hazardous emission problems associated with current CHP solutions, thus making CHP solutions more environmentally acceptable to produce, transport and use. In addition, the CHP-diluent solution should have minimal or no detrimental effects on the end user's finished product or process yield(s). The CHP-diluent solution should also preferably depress the CHP solution's freezing point to obviate the necessity of CHP users having to switch concentrations of CHP in response to changing climatic conditions at their facilities. Moreover, the diluent should have minimal effect on the viscosity of the resultant solution.

### Summary of the Invention

The present invention provides a method of reducing hazardous emissions from a purified cumene hydroperoxide solution by adding from 1 to 6 weight percent water to produce a mixture. The present invention further provides a method of depressing the freezing point of a purified cumene hydroperoxide solution by adding from 1 to 6 weight percent water to produce a mixture. The present invention still further provides for a composition comprising cumene hydroperoxide solution with from 1 to 6 weight percent water. The present invention yet further provides a method of preparing crude CHP solution for transport by providing crude CHP solution; purifying the crude CHP to a purity of at least 80%; producing a mixture by adding from 1 to 6 weight percent water to the purified CHP solution and placing the mixture in a transport vessel. The present invention yet still further provides a method of obviating the seasonal adjustment of CHP concentrations in transporting purified CHP solutions by adopting a standard concentration CHP solution; adding from 1 to 6 weight percent water to the standard concentration CHP solution to produce a mixture and shipping the mixture at all times of the year. These and other advantages and benefits will be apparent from the Detailed Description of the Invention herein below.

### Brief Description of the Figures

The present invention will be described for the purposes of illustration, but not limitation, in conjunction with the following figures, wherein:

Figure 1 illustrates a CHP-Water Assay using approximately 88% CHP;

Figure 2 illustrates a CHP-Water Assay using approximately 90% CHP;

Figure 3 illustrates freezing point depression data for various CHP-water blends; and

Figure 4 illustrates viscosity of CHP-water solutions at various temperatures.

## Detailed Description of the Invention

The method of the present invention involves the use of water, from about 1 to about 6 weight percent, as a diluent for purified CHP solutions. By purified CHP solutions the inventors herein mean a solution which prior to dilution is greater than 80% CHP with the remainder being predominately dimethyl benzylalcohol and acetophenone. It most likely has undergone some purification to remove nearly all the considerably lower-boiling and the high boiling impurities.

In addition to being considered an impurity, water is generally thought, by those of ordinary skill in the art, to have little solubility in CHP and therefore of no practical significance. Contrary to this accepted wisdom, the Inventors have found that water can be added to purified CHP solutions as an alternative diluent for a portion of or all of the previously added cumene. Although it is well known that solvents tend to depress the freezing point of materials, the Inventors did not expect such a small percentage of water to so significantly depress the freezing point of CHP solutions. One of ordinary skill in the art would have expected cumene to have a greater effect than water on freezing points depression because the freezing point range of the CHP solutions is well below the freezing point of water. The freezing point of cumene itself is  $-96^{\circ}\text{C}$ . Water is known to be used in the prior art as a diluent for organic substances that are highly soluble in water and have melting points well above the freezing point of water, such as phenol and neopentyl glycol.

The Inventors have found that water can be used as a diluent to reduce the CHP concentration to levels which will comply with the United States regulations for the transportation of CHP. Such use of water, rather than cumene, as a diluent provides several advantages in the resultant CHP solution. One beneficial effect of the use of water as a diluent is to reduce the hazardous emissions encountered in prior art CHP solutions because a hazardous chemical, cumene, is not added to the purified CHP solution. Another beneficial effect of water as a diluent for CHP solutions is that water will depress the freezing point of the resultant solution by as much as  $15^{\circ}$  to  $20^{\circ}\text{F}$  ( $8.4$  to  $11.2^{\circ}\text{C}$ ). This lowered freezing point is advantageous for several reasons.

1. A higher purity CHP solution can be utilized by end users on a year-round basis because the freezing point of the water-diluted CHP solution is lowered. This should significantly reduce or eliminate the end user's need to change CHP concentrations in response to changes in ambient temperatures at their facilities.
2. The addition of water to purified CHP solutions should also reduce cumene-related impurities in the end user's finished products
3. The addition of water to purified CHP solutions may increase process yields.
4. The suppliers of CHP-water solutions may save money by standardizing their production facilities, because they may not have to dilute purified CHP solutions to different percentages at various times of the year depending on climatic conditions at end user's facilities. In addition, the suppliers will not have to add cumene to their purified CHP solutions, thereby

reducing both the environmental hazards and the production costs associated with making CHP solutions.

Because of the limited solubility of water in CHP, an excessive amount of water added to the CHP will form a second aqueous phase in the container. Therefore, a reliable method of assaying the amount of water in the CHP phase of the solution was developed.

#### Water Assay for CHP

An empty bottle was weighed to obtain a tare weight. An aliquot of purified CHP, of either approximately 88% or 90% purity, was added and the bottle's weight re-determined. Subtracting the tare weight from the weight of the bottle with purified CHP yielded the net weight of purified CHP. An aliquot of water was added to the bottle. The aliquot was approximately the amount needed to produce the desired percentage of water added for the sample, i.e., 1%, 2%, etc. The bottle's weight was again determined and this weight subtracted from the weight of the bottle with purified CHP, thus yielding a net weight of water. The net weight of water was divided by the net weight of purified CHP to yield the percent water actually added. The CHP-water mixtures were shaken until clear.

One ml CHP-water samples, taken from CHP-water solutions prepared as above, were injected into a gas chromatograph (Hewlett-Packard model HP 5890) equipped with a thermal conductivity detector, and a 6 ft. x 1/8 inch (182.88 cm x 0.318 cm) stainless steel capillary column containing 80-100 mesh Porapack-Q® packing material manufactured by Supelco and others. The chromatograph was operated at a temperature below 220°C with a flow rate of 20 ml/min column and 30 ml/min reference helium. A typical chromatogram shows the water peak eluting at about 2.7 minutes and the CHP between 4.5 and 5.5 minutes. The area count is converted to a weight percent based on calibration of external standards with known amounts of water addition to acetone. For this method, the Inventors prefer to use an auto-sampler for precise and reproducible control of amount injected.

The results of the CHP-water analysis are contained in Table I and illustrated in Figure 1 for purified CHP with an approximate purity of 88% and in Table II and Figure 2 for purified CHP with an approximate purity of 90%. Analysis of the starting material indicated that a slight amount of water was present. It will be apparent from comparing the data and looking at the figures, that this assay is accurate and reproducible because the amounts determined by both trials in this assay correlate rather well to the actual amounts of water added, less that initially present, and to each other for 88% CHP and 90% CHP. The higher concentrations of water in purified CHP solutions appear to produce a greater deviation between the actual amounts of water added and that analyzed than at lower water concentrations. Without being limited to any specific explanation for this phenomenon, the Inventors believe that it is more difficult to completely dissolve water in CHP as the solution approaches the solubility limit of water in CHP.



**TABLE I – Analysis of 88% CHP + Water Samples**

Sample No.	Contents 88% CHP	Tare Wt	Tare Wt + CHP	Net Wt CHP	Tare Wt + CHP + Water	Net wt Water	Water Added (Actual) weight percent	Water Added (Determined by CHP-water Assay) weight percent	
		g	g	g	g	g		Trial A	Trial B
1	+0% water	15.22	31.74	16.51	31.74	0.00	0.00	0.60	0.62
2	+1% water	14.95	31.02	16.07	31.19	0.16	1.01	1.59	1.58
3	+2% water	14.99	30.78	15.78	31.11	0.33	2.10	2.56	2.46
4	+3% water	15.19	30.77	15.57	31.25	0.48	3.09	3.47	3.39
5	+4% water	15.06	31.04	15.97	31.67	0.63	3.95	4.18	4.21
6	+5% water	15.11	31.39	16.27	32.22	0.83	5.10	5.25	5.34
7	+6% water	15.12	31.44	16.32	32.44	1.00	6.12	5.54	5.62

**TABLE II – Analysis of 90% CHP + Water Samples**

Sample No.	Contents 90% CHP	Tare Wt	Tare Wt + CHP	Net Wt CHP	Tare Wt + CHP + Water	Net wt Water	Water Added (Actual) weight percent	Water Added (Determined by CHP-water Assay) weight percent	
		g	g	g	g	g		Trial A	Trial B
1	+0% water	15.23	31.89	16.66	31.89	0.00	0.00	0.68	0.80
2	+1% water	15.08	31.63	16.55	31.80	0.17	1.02	1.64	1.59
3	+2% water	15.13	31.67	16.55	32.01	0.34	2.05	2.55	2.57
4	+3% water	15.10	31.95	16.85	32.43	0.48	2.87	3.38	3.38
5	+4% water	15.22	32.05	16.83	32.70	0.65	3.88	4.21	4.27
6	+5% water	15.04	32.52	17.47	33.38	0.86	4.94	5.23	5.28
7	+6% water	15.17	32.42	17.26	33.46	1.03	5.98	6.02	6.04

**Freezing Point Test**

The effect of water as a diluent on the freezing point of various CHP-water solutions was determined and the results are summarized in Table III. Briefly, production samples of CHP diluted with from 1 weight percent to 6 weight percent water were frozen in a Thermotron® cooling chamber (Venturedyne Ltd., Milwaukee, WI, USA) equipped with an internal temperature controller and an external thermocouple temperature meter. A boiling chip was added to each sample to reduce supercooling because of the difficulty in freezing small samples of CHP. The material becomes very

viscous and requires a nucleating site to begin forming a white crystalline solid. All samples were cooled to about -37°F (-38°C). After all the samples were frozen, the cooling chamber temperature was increased in 1-2°F (0.56 –1.11°C) increments and stabilized for a minimum of four hours. The condition of the samples was visually observed and recorded. Table III lists the temperature at which the sample appeared to contain only liquid, i.e., the melting or freezing point. Assessment of the samples containing 5% and 6 % water was complicated by the fact that some of the water formed a separate phase which then froze into ice crystals on the side of the vial. These ice crystals made it difficult to determine exactly when the rest of the liquid was completely melted. The data in the table are the best estimates of when essentially all of the liquid had melted.

**TABLE III – Melting Point for Various CHP-Water Solutions**

Wt.% H <sub>2</sub> O	Temperature at Which Sample First Appeared to all Liquid						
	20 ml Bottles		1 ml Vials		1 ml Vials		20 ml Vial
	88% CHP °F (°C)	90%CHP °F (°C)	88%-Trial A °F (°C)	88%-Trial B °F (°C)	90%-Trial A °F (°C)	90%-Trial B °F (°C)	91.9% CHP °F (°C)
0	+1.0 (-17.2)	+6.3 (-14.3)	-3.7 (-19.8)	-3.7 (-19.8)	-2.2 (-19.0)	-2.2 (-19.0)	
1	-9.0 (-22.8)	-2.2 (-19.0)	-11.0 (-23.9)	-11.0 (-23.9)	-11.0 (-23.9)	-11.0 (-23.9)	
2	-16.0 (-26.7)	-11.0 (-23.9)	-17.0 (-27.2)	-17.0 (-27.2)	#	#	+2.2 (-16.6)
3	-16.0 (-26.7)	-17.0 (-27.2)	-16.0 (-26.7)	-16.0 (-26.7)	-17.7 (-27.6)	-17.7 (-27.6)	-1.2 (-18.4)
4	-2.2 (-19.0)	-2.2* (-19.0)	-9.0 (-22.8)	-9.0 (-22.8)	-9.0 (-22.8)	-9.0 (-22.8)	-3.0 (-19.4)
5	-2.2* (-19.0)	-2.2* (-19.0)	11.7* (-11.3)	11.7* (-11.3)	6.3* (-14.3)	6.3* (-14.3)	
6	-2.2* (-19.0)	-2.2* (-19.0)	11.7* (-11.3)	11.7* (-11.3)	14.6* (-9.7)	14.6* (-9.7)	

# - Never froze

\* - Essentially all liquid, some ice crystals on container's inside wall up to 32°F, 0°C

In the interest of clarity, only data from the experiments conducted using 20 ml bottles are shown in Figure 3. As can be seen from the data in Table III and by reference to Figure 3, the greatest freezing point depression of both 88% and 90% purity CHP solutions occurs with the use of between 2 to 3 weight percent water as a diluent. It will be seen from comparison of this data with data obtained by using 1 ml vials at each purity of CHP solution that the maximum freezing point depression is consistently observed with the use of between 2 to 3 weight percent water as a diluent. In Table III, the addition of 2% cumene in 90% CHP lowered the melt point from +6.3°F (-14.3°C) to +1.0°F (-17.2°C) while adding 2% water lowered the melt point from +6.3°F (-14.3°C) to -11.0°F (-23.9°C), thus demonstrating the unexpected superiority of water over cumene at lowering the freezing point.

### **Viscosity Test**

Because the viscosity of the CHP-water solution at lower temperatures may have an effect on its suitability for end users, the viscosity of CHP-water solutions at various temperatures was determined by the following test.

A beaker with a 500 ml CHP-water sample was cooled in an acetone/dry ice bath. As the sample warmed, the viscosity was measured with a Brookfield™ LVT Viscometer with a No. 1 or No. 2 spindle at two speeds between 12 and 60 rpm. The viscosity in centipoise (cp) at two speeds was averaged and recorded. The temperatures were measured with a thermocouple and a meter. Temperatures were not exact as there was a constant drift upward in temperature; the average over the measuring period for that sample was reported. The results are contained in Table IV, and are illustrated in Figure 4. As will be apparent from a review of Table IV and Figure 4, the addition of 2 weight percent water to an approximately 90% purity CHP solution appears to have very little effect on viscosity over the temperature ranges assayed. The addition of 2 weight percent water as a diluent of 91.9% purity CHP does slightly increase viscosity of the solution at lower temperatures over the viscosity seen in 90% CHP alone or 90% + 2 weight percent water.

**Table IV      CHP-Water Solutions Viscosity Measurements**

<u>Temp °F (°C)</u>	<u>90% CHP + 0</u> <u>weight percent</u> <u>Water</u> <u>Viscosity (cp)</u>	<u>90% CHP + 2 weight</u> <u>percent Water</u> <u>Viscosity (cp)</u>	<u>91.9% CHP + 2 weight</u> <u>percent Water</u> <u>Viscosity (cp)</u>
77.1 (25.1)		13.0	13.1
76.7 (24.8)	13.2		
63.0 (17.2)	19.6	19.0	20.3
57.7 (14.3)	22.5		
50.3 (10.2)		28.0	
48.6 (9.2)	33.6		31.4
44.0 (6.7)	34.5		
35.0 (1.7)	57.5		
34.2 (1.2)		57.0	
33.2 (0.7)	61.5		
31.0 (-0.6)	65.3		
30.6 (-0.8)	67.0	68.5	
29.2 (-1.6)	73.0		
27.9 (-2.3)	80.2		
23.2 (-4.9)	88.8	94.0	
22.3 (-5.4)			102.5
14.2 (-9.9)			199.8
11.2 (-11.6)		190.0	
9.8 (-12.3)	227.5		325.0
7.2 (-13.8)			306.0
5.0 (-15.0)	311.0	300.0	
2.0 (-16.7)			535.0
0.0 (-17.8)	363.0		
-2.0 (-18.9)	450.0		
-2.9 (-19.4)			710.0
-4.0 (-20.0)		690.0	

5            Although the description of the invention herein utilized small bottles, the Inventors contemplate the use of much larger vessels on a commercial scale, including but not limited to, large tanks for adding

water to the purified CHP solutions and drums, tank trucks, tank cars or pipelines for transporting the CHP-water mixtures to end users.

The foregoing illustrations of embodiments of the present invention are offered for the purposes of illustration and not limitation. It will be readily apparent to those skilled in the art that the 5 embodiments described herein may be modified or revised in various ways without departing from the spirit and scope of the invention. The scope of the invention is to be measured by the appended claims.

## CLAIMS

We claim:

1. A method of reducing hazardous emissions from a purified cumene hydroperoxide (CHP) solution, said method comprising diluting said purified CHP solution with x weight percent water to produce a mixture;  
wherein  $1 \leq x \leq 6$ .
2. The method of claim 1 further including mixing said CHP-water mixture.
3. The method of claim 1 further including transporting said CHP-water mixture.
4. The method of claim 1, wherein  $2 \leq x \leq 3$ .
5. The method of claim 1, wherein said purified CHP solution is at least 80% CHP.
6. The method of claim 1, wherein said purified CHP solution is at least 88% CHP.
7. The method of claim 1, wherein said purified CHP solution is at least 90% CHP.
8. A method of depressing the freezing point of a purified cumene hydroperoxide (CHP) solution, said method comprising diluting said purified CHP solution with x weight percent water to produce a CHP mixture;  
wherein  $1 \leq x \leq 4$ .
9. The method of claim 8 further including mixing said CHP-water mixture.
10. The method of claim 8 further including transporting said CHP-water mixture.
11. The method of claim 8, wherein  $2 \leq x \leq 3$ .
12. The method of claim 8, wherein said purified CHP solution is at least 80% CHP.
13. The method of claim 8, wherein said purified CHP solution is at least 88% CHP.
14. The method of claim 8, wherein said purified CHP solution is at least 90% CHP.
15. A composition comprising:  
purified cumene hydroperoxide (CHP); and  
x weight percent water  
wherein  $1 \leq x \leq 6$ .
16. The composition of claim 15, wherein  $2 \leq x \leq 3$ .

17. The composition of claim 15, wherein said purified CHP solution is at least 80% CHP.

18. The composition of claim 15, wherein said purified CHP solution is at least 88% CHP.

19. The composition of claim 15, wherein said purified CHP solution is at least 90% CHP.

20. A method of preparing a crude cumene hydroperoxide (CHP) solution for transport, said  
5 method comprising:

providing crude CHP solution;

purifying said crude CHP solution to at least 80% CHP; and

producing a CHP-water mixture by adding x weight percent water to the purified CHP  
solution,

10 wherein

$$0 < x \leq 6.$$

21. The method of claim 20 further including transporting said CHP-water mixture.

22. The method of claim 20 wherein  $2 < x \leq 3$ .

23. A method of obviating the seasonal adjustment of cumene hydroperoxide (CHP)  
15 concentration in transporting purified CHP solutions, said method comprising:

adopting a standard concentration CHP solution;

producing a CHP-water mixture by adding x weight percent water to said standard  
concentration CHP

solution, wherein  $1 \leq x \leq 6$ ; and

20 transporting said mixture at all times of the year.

24. The method of claim 23, wherein said standard concentration CHP solution is at least 80%  
CHP.

25. The method of claim 23, wherein said standard concentration CHP solution is at least 88%  
CHP.

26. The method of claim 23, wherein said standard concentration CHP solution is at least 90%  
25 CHP.

27. The method of claim 23, wherein  $2 \leq x \leq 3$ .

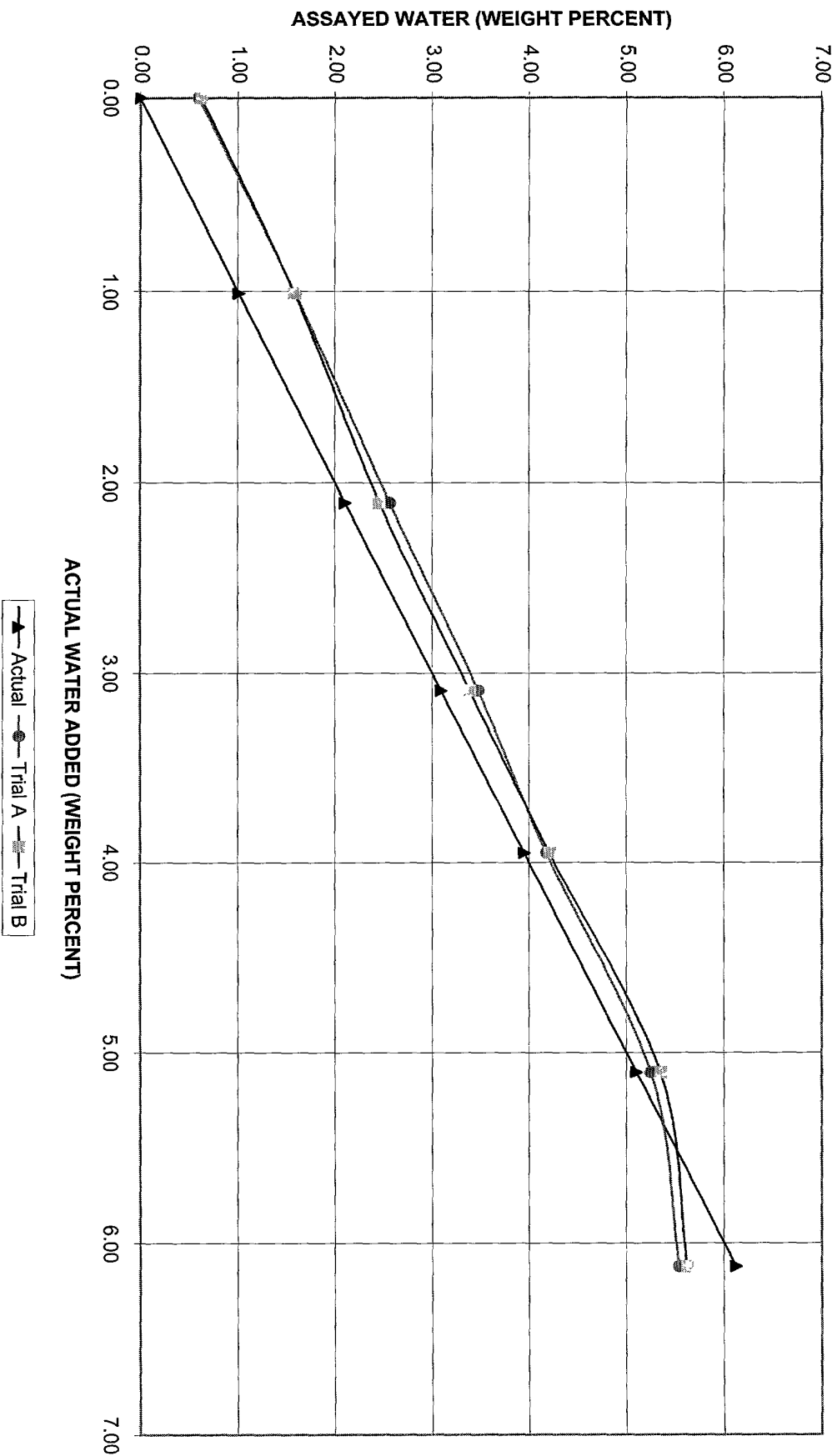
## ABSTRACT

The present invention provides for the use of water, rather than cumene, as a more environmentally acceptable diluent for purified cumene hydroperoxide (CHP) solutions. From 1 to about 5 6 weight percent water can be used to dilute purified CHP solutions, thus reducing or eliminating the use of a hazardous compound, cumene, as a diluent. The method and CHP-water solutions of the present invention should significantly reduce or eliminate the hazardous emissions problems encountered with the use of cumene as a diluent and make CHP solutions more environmentally acceptable to produce, transport and use. Water as a diluent also depresses the freezing point of the resultant solution, thereby 10 permitting year-round use of higher concentration CHP solutions. Water diluted CHP solutions will also reduce cumene-related impurities in finished products made therefrom.



Figure 1

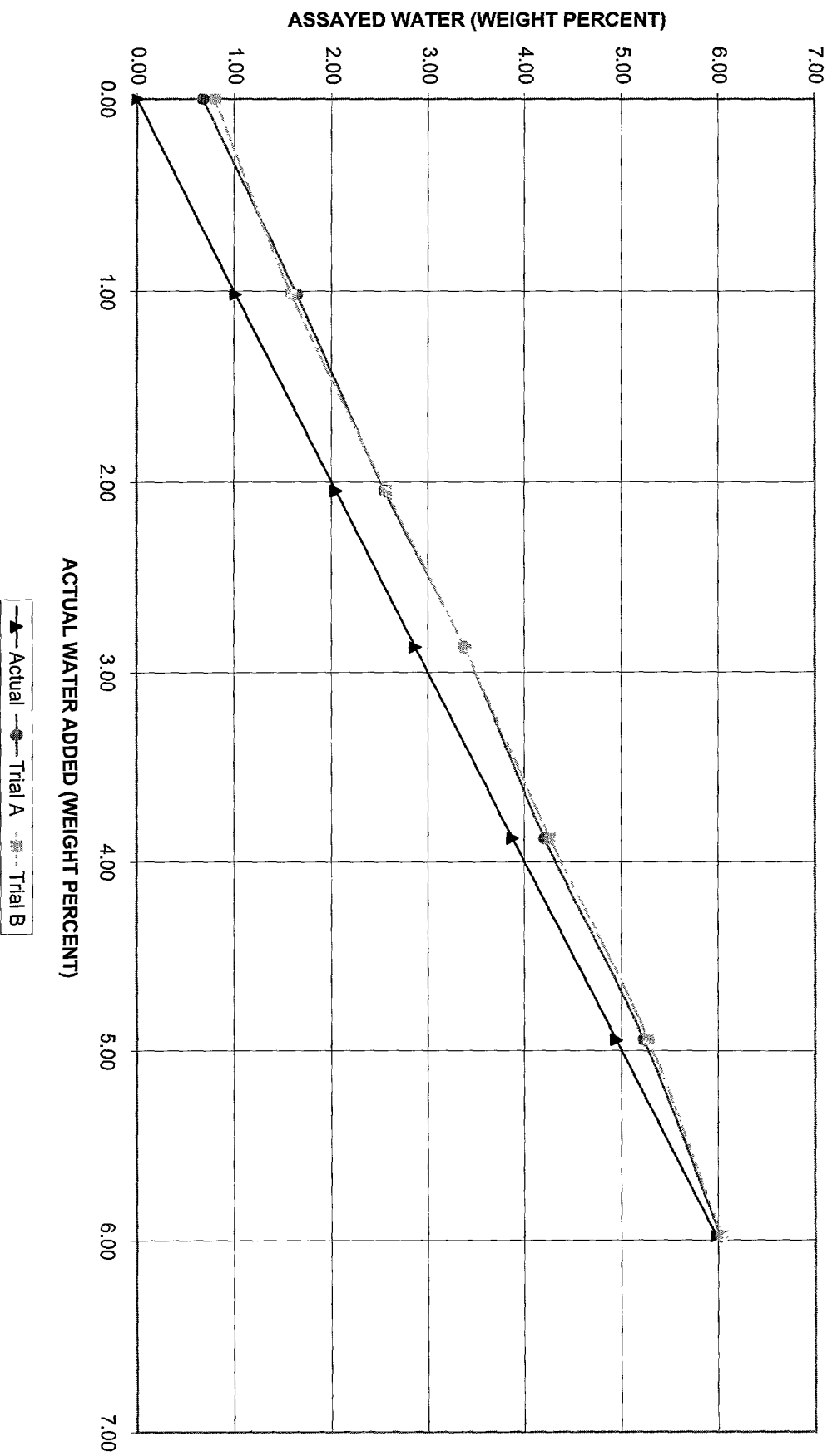
ASSAYED VS. ACTUAL WATER ADDED (88% CHP)



09650099 094200

Figure 2

ASSAYED VS. ACTUAL WATER ADDED (90% CHP)



09650099.094200

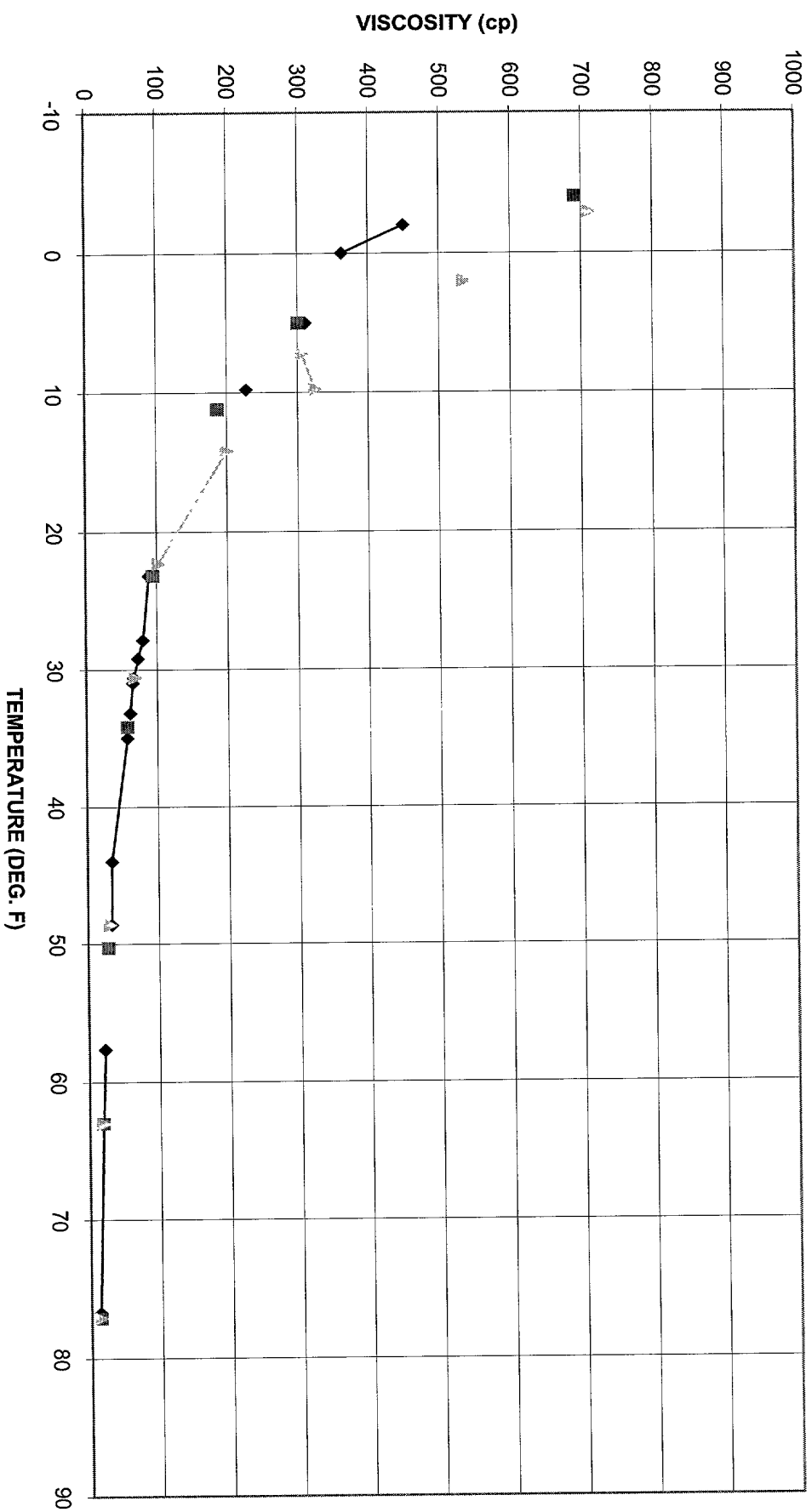
## MELT POINT FOR VARIOUS CHP/WATER BLENDS



Parameter	Value	Unit	Source
$\alpha_1$	0.0000		
$\alpha_2$	0.0000		
$\alpha_3$	0.0000		
$\alpha_4$	0.0000		
$\alpha_5$	0.0000		
$\alpha_6$	0.0000		
$\alpha_7$	0.0000		
$\alpha_8$	0.0000		
$\alpha_9$	0.0000		
$\alpha_{10}$	0.0000		
$\alpha_{11}$	0.0000		
$\alpha_{12}$	0.0000		
$\alpha_{13}$	0.0000		
$\alpha_{14}$	0.0000		
$\alpha_{15}$	0.0000		
$\alpha_{16}$	0.0000		
$\alpha_{17}$	0.0000		
$\alpha_{18}$	0.0000		
$\alpha_{19}$	0.0000		
$\alpha_{20}$	0.0000		
$\alpha_{21}$	0.0000		
$\alpha_{22}$	0.0000		
$\alpha_{23}$	0.0000		
$\alpha_{24}$	0.0000		
$\alpha_{25}$	0.0000		
$\alpha_{26}$	0.0000		
$\alpha_{27}$	0.0000		
$\alpha_{28}$	0.0000		
$\alpha_{29}$	0.0000		
$\alpha_{30}$	0.0000		
$\alpha_{31}$	0.0000		
$\alpha_{32}$	0.0000		
$\alpha_{33}$	0.0000		
$\alpha_{34}$	0.0000		
$\alpha_{35}$	0.0000		
$\alpha_{36}$	0.0000		
$\alpha_{37}$	0.0000		
$\alpha_{38}$	0.0000		
$\alpha_{39}$	0.0000		
$\alpha_{40}$	0.0000		
$\alpha_{41}$	0.0000		
$\alpha_{42}$	0.0000		
$\alpha_{43}$	0.0000		
$\alpha_{44}$	0.0000		
$\alpha_{45}$	0.0000		
$\alpha_{46}$	0.0000		
$\alpha_{47}$	0.0000		
$\alpha_{48}$	0.0000		
$\alpha_{49}$	0.0000		
$\alpha_{50}$	0.0000		
$\alpha_{51}$	0.0000		
$\alpha_{52}$	0.0000		
$\alpha_{53}$	0.0000		
$\alpha_{54}$	0.0000		
$\alpha_{55}$	0.0000		
$\alpha_{56}$	0.0000		
$\alpha_{57}$	0.0000		
$\alpha_{58}$	0.0000		
$\alpha_{59}$	0.0000		
$\alpha_{60}$	0.0000		
$\alpha_{61}$	0.0000		
$\alpha_{62}$	0.0000		
$\alpha_{63}$	0.0000		
$\alpha_{64}$	0.0000		
$\alpha_{65}$	0.0000		
$\alpha_{66}$	0.0000		
$\alpha_{67}$	0.0000		
$\alpha_{68}$	0.0000		
$\alpha_{69}$	0.0000		
$\alpha_{70}$	0.0000		
$\alpha_{71}$	0.0000		
$\alpha_{72}$	0.0000		
$\alpha_{73}$	0.0000		
$\alpha_{74}$	0.0000		
$\alpha_{75}$	0.0000		
$\alpha_{76}$	0.0000		
$\alpha_{77}$	0.0000		
$\alpha_{78}$	0.0000		
$\alpha_{79}$	0.0000		
$\alpha_{80}$	0.0000		
$\alpha_{81}$	0.0000		
$\alpha_{82}$	0.0000		
$\alpha_{83}$	0.0000		
$\alpha_{84}$	0.0000		
$\alpha_{85}$	0.0000		
$\alpha_{86}$	0.0000		
$\alpha_{87}$	0.0000		
$\alpha_{88}$	0.0000		
$\alpha_{89}$	0.0000		
$\alpha_{90}$	0.0000		
$\alpha_{91}$	0.0000		
$\alpha_{92}$	0.0000		
$\alpha_{93}$	0.0000		
$\alpha_{94}$	0.0000		
$\alpha_{95}</$			

Figure 4

VISCOSITY OF CHP/WATER BLENDS



00560099 : 004200

Please type a plus sign (+) inside this box  $\div$  +

PTO/SB/01 (12-97)

Approved for use through 9/30/00. OMB 0651-0032  
Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

<b>DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION (37 CFR 1.63)</b>	<b>Attorney Docket Number</b>		DB000817-000	
	<b>First Named Inventor</b>		Henry, Keith E.	
	<i>COMPLETE IF KNOWN</i>			
	<b>Application Number</b>	Not yet assigned		
	<b>Filing Date</b>			
	<b>Group Art Unit</b>	Not yet assigned		
<input checked="" type="checkbox"/> Declaration Submitted with Initial Filing	OR	<input type="checkbox"/> Declaration Submitted after Initial Filing (surcharge (37 CFR 1.16 (e)) required)	<b>Examiner Name</b>	Not yet assigned

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

WATER DILUTED CUMENE HYDROPEROXIDE SOLUTIONS

the specification of which

(Title of the Invention)

☒ is attached hereto  
OR

☐ was filed on (MM/DD/YYYY)  as United States Application Number or PCT International

Application Number  and was amended on (MM/DD/YYYY)  (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or 356(b) of any foreign application(s) for patent or inventor's certificate, or 356(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached?	
				YES	NO
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

☐ Additional foreign application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto:

I hereby claim the benefit under 35 U.S.C. 119(e) of any United States provisional application(s) listed below.

Application Number(s)	Filing Date (MM/DD/YYYY)	<input type="checkbox"/> Additional provisional application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto.

[Page 1 of 2]

Burden Hour Statement: This form is estimated to take 0.4 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

Please type a plus son (+) inside this box  $\div$  +

PTO/SB/01 (12-97)  
Approved for use through 9/30/00. OMB 0651-0032  
Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

## DECLARATION — Utility or Design Patent Application

I hereby claim the benefit under 35 U.S.C. 120 of any United States application(s), or 365(c) of any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

U.S. Parent Application or PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (if applicable)

☐ Additional U.S. or PCT international application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto.

As a named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

☐ Customer Number  

OR

☐ Registered practitioner(s) name/registration number listed below

Place Customer  
Number Bar Code  
Label here

Name	Registration Number	Name	Registration Number

☐ Additional registered practitioner(s) named on supplemental Registered Practitioner Information sheet PTO/SB/02C attached hereto.

Direct all correspondence to:

☐ Customer Number  
or Bar Code Label  

OR ☒ Correspondence address below

Name					
Address					
Address					
City		State		ZIP	
Country		Telephone		Fax	

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name of Sole or First Inventor:

☐ A petition has been filed for this unsigned inventor

Given Name (first and middle [if any])		Family Name or Surname	
Keith E.		Henry	
Inventor's Signature	<i>Keith E. Henry</i>		Date
Residence: City	Allison Park	State	PA
Country	United States	Citizenship	United States
Post Office Address	4705 Middle Road		
Post Office Address			
City	Allison Park	State	PA
ZIP	15101-1115	Country	United States

☒ Additional inventors are being named on the 1 supplemental Additional Inventor(s) sheet(s) PTO/SB/02A attached hereto

Please type a plus sign (+) inside this box → +Approved for use through 9/30/98. OMB 0651-0032  
Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

**DECLARATION****ADDITIONAL INVENTOR(S)  
Supplemental Sheet**Page 1 of 1**Name of Additional Joint Inventor, if any:**☐ A petition has been filed for this unsigned inventor

Given Name (first and middle [if any])

Family Name or Surname

John E.

Aiken

Inventor's  
Signature*John E. Aiken*9/8/00  
Date

Residence: City

Monroeville

State

PA

Country

United States

Citizenship

United  
States

Post Office Address

207 Greenvale Drive

Post Office Address

City

Monroeville

State

PA

ZIP

15146

Country

United States

**Name of Additional Joint Inventor, if any:**☐ A petition has been filed for this unsigned inventor

Given Name (first and middle [if any])

Family Name or Surname

Inventor's  
Signature

Date

Residence: City

State

Country

Citizenship

Post Office Address

Post Office Address

City

State

ZIP

Country

**Name of Additional Joint Inventor, if any:**☐ A petition has been filed for this unsigned inventor

Given Name (first and middle [if any])

Family Name or Surname

Inventor's  
Signature

Date

Residence: City

State

Country

Citizenship

Post Office Address

Post Office Address

City

State

ZIP

Country

Burden Hour Statement: This form is estimated to take 0.4 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.